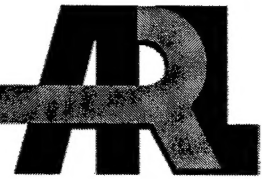ARMY RESEARCH LABORATORY

Induction Curing of a Phase-Toughened Adhesive

by Christian J. Yungwirth, Eric D. Wetzel, and James M. Sands

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Eric D. Wetzel and James M. Sands
Weapons and Materials Research Directorate, ARL

Christian J. Yungwirth
Oak Ridge Institute for Science and Education

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*Christian is a research associate for the Oak Ridge Institute for Science and Technology, Oak Ridge, TN.

14. ABSTRACT

Phase-toughened epoxy/dicyandiamide adhesives were loaded with magnetic particle filler and then cured by induction processing. The effect of induction field magnitude and exposure time on bonding strength of the loaded adhesives was characterized. At low magnetic field amplitudes, bond strength increased significantly with exposure time, with the highest strength bonds occurring after 60-min exposures. At high magnetic field amplitudes, bond strength increased only slightly with exposure time, so that significant bond strength was reached after 15 min of exposure. In general, the induction-cured adhesives exhibited lower bond strengths than comparable oven-cured adhesives. All of these strength trends were likely due to process-induced variations in the adhesive degree of cure, toughening phase development, or thermal degradation.

15. SUBJECT TERMS

induction curing, phase-toughened adhesive, dicyandiamide, magnetic powders, epoxy, magnetic susceptor, DICY

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1. Introduction

1.1 Motivation

The U.S. Army has identified a need for lightening its forces in order to improve their deployability, survivability, and lethality. This lightening requires the integration of new materials, including polymer matrix composite materials.

A significant challenge to implementation of these new materials is joining or bonding of composite structures (1, 2). During manufacturing, bonding methods are needed which are repeatable, rapid, and low cost. Joining is also critical for repair or replacement of damaged structures, a concern that is particularly relevant to Army structures.

1.2 Conventional Bonding Methods

There are two conventional approaches for joining polymer matrix composites—mechanical fasteners and surface bonding techniques.

Mechanical fasteners are the most basic joining method. However, mechanical fasteners also introduce stress concentration points which limit structural performance (3). Additionally, the weight penalty associated with mechanical fasteners is a detriment to the construction of lightweight structures.

Surface bonding techniques are preferred to mechanical fasteners primarily due to their superior load transfer characteristics. For high-performance engineering applications, surface bonding is typically achieved using elevated temperature-cure, thermosetting adhesives. These thermosetting adhesives usually require temperatures of 120–200 °C for 5–120 min to complete the bond (4).

The most common ways of heating adhesive bondlines are convection ovens, thermal blankets, and radiant heaters. Convection ovens work by heating the surrounding air, which then transfers heat to the adherend. Thermal blankets heat the adherend by direct contact. Radiant heaters transfer their energy to the adherend via infrared radiation. All of these processes heat the outer surface of the adherend, and the heat is then conducted to the bondline. The extra time and energy associated with heating the adherends, rather than heating the bondline directly, reduces the overall process efficiency. Additionally, for composite materials, the cure temperature of certain adhesive systems may be near the degradation limits of the adherends. In this case, longer adhesive cure cycles increase the likelihood of adherend thermal degradation.

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1.3 Induction Heating

Induction heating works by exposing a conductive or magnetic material to a high-frequency electromagnetic field, usually between 50 kHz and 100 MHz. Any material that heats up when exposed to an electromagnetic field is called a "susceptor" material. The electromagnetic field can induce heating through two mechanisms. If the susceptor material is conductive, then eddy currents are induced in the conductor, and the conductor will then heat due to resistive effects. If the material is magnetic, hysteresis losses from the magnetization-demagnetization process cause additional heating. This mechanism of heating is called hysteresis heating.

Induction heating can be used for adhesive curing if a susceptor material is embedded in the bondline and the adherends are nonsusceptors. In this case, an incident induction field will penetrate through the adherends and directly heat the bondline, enabling adhesive cure. The primary advantage of this approach is that energy is directly coupled into the bondline, enabling rapid processing of embedded bondlines.

Two general types of adhesive susceptors have been previously investigated. The first type is a metal screen located within the bondline (5). Once exposed to the electromagnetic field, the screen begins to heat due to resistive heating. The limitation of this approach is that the heating is unbounded and usually nonuniform. This lack of homogeneity may lead to regions of adhesive which are undercured or thermally degraded. Although these problems can sometimes be mitigated through the use of temperature sensors and active induction field control, the associated processing complexities can be prohibitive.

The second type of adhesive susceptor is magnetic powder with Curie temperature-limited heating (6, 7). These materials use hysteresis heating and, if all other heating effects are dominated by the hysteresis heating, will not heat beyond their Curie temperature. The benefit of this approach is that magnetic powders can be chosen whose Curie temperature can be matched to the desired processing temperature of the adhesive system (8–10). If the electromagnetic field is powerful enough to maintain this hysteresis heating, then the adhesive will dwell at this temperature for as long as it is exposed to the field (11). This feature greatly improves bondline temperature control and uniformity, without the use of sensors or complex controls.

In this report, we demonstrate the use of induction heating of magnetic powder susceptors for thermal curing of adhesive systems and investigate whether induction heating can reduce process times compared with traditional heating methods.

1.4 Toughened Adhesives

The strength of a thermosetting adhesive is determined by two different mechanisms—network formation and toughening effects. Network formation refers to the degree of completion of cure, with strength increasing continuously as the degree of cure approaches 100% conversion of reactive groups. In general, unmodified thermoset epoxies and acrylic-type adhesives are relatively brittle and fail under relatively low stress conditions. Therefore, various toughening modifications are commonly designed into thermosetting adhesives.

A phase-toughened adhesive contains rubber particles, which improve the adhesive's fracture toughness. Adhesive failure typically occurs due to crack formation and propagation. If a crack reaches a rubber particle, the low modulus of the rubber phase reduces the local stress concentration at the crack tip, slowing the growth rate. As particle size decreases (at a fixed volume fraction), the dispersion of particles throughout the adhesive matrix is improved, increasing their toughening effect. However, if the rubber particles are too small, their toughening effect is negligible. Therefore, there is an optimal particle size for achieving maximum adhesive toughness (12).

There are two traditional approaches to incorporating secondary particles in an adhesive. The first approach, the one used in our study, is to formulate the adhesive to develop particles during the cure process through phase separation. Typically, the secondary phase is a modified rubber or thermoplastic that has a high compatibility with the uncured monomer of the adhesive. However, during cure, the adhesive network begins to cross-link, and the rubber groups coalesce and separate from the forming network due to decreases in favorable mixing (13). The particle size continuously grows during cure until molecular motion is limited by vitrification. Since the processes of monomer aggregation and network formation have independent kinetic characteristics, different processing histories result in different sizes and numbers of equilibrium particle domains. Additionally, very rapid processing can result in trapping of the secondary tougheners in the matrix and create decreased matrix performance characteristics both thermally and mechanically.

The second toughening approach is adding preformed particles to the adhesive a priori. In this approach, the optimal size of the particles is selected prior to curing and is well controlled. The most significant drawback of this approach is that the adhesive viscosity dramatically increases, making handling difficult and often requiring a change in formulation to improve substrate wetting and bonding. Additionally, the preformed particles perform less efficiently than particles formed in situ (14).

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2. Experimental

2.1 Materials and Equipment

2.1.1 Adhesive

The adhesive system chosen was a dicyandiamide (DICY)-cured epoxy that was epoxy-terminated butadiene-acrylonitrile phase toughened. We will refer to this adhesive system as D1. This adhesive was chosen for its excellent mechanical properties, phase separation characteristics, and cure temperature, which is similar to the dwell temperatures of the magnetic particles.

The adhesive formulation was environmentally stable for room temperature storage, providing a working life of at least 1 year. The cured adhesive possessed the generally desired characteristics of high thermal stability (250 °F), good solvent resistance, and low moisture uptake.

2.1.2 Magnetic Particles

The magnetic susceptor particles chosen were FP160 nickel zinc ferrite (PowderTech Corp., Valparaiso, IN), a magnetically soft ferrite. Although the particles did not have a sharp Curie temperature, the magnetization did diminish gradually with temperature. The saturation magnetization of the particles was ~0 (nonmagnetic) by 250 °C, producing a limiting condition on heating.

As-received magnetic particles were ball milled for 3 hr prior to the addition of the D1 adhesive. This step reduced the particle size to ~10–100 μm in diameter.

In order to minimize the impact of particle settling in the adhesive matrix during processing and storage, the milled FP160 was coated with a reactive surface modifier. The modifier selected was (3-glycidoxypropyl) trimethoxysilane, which was added to the dry FP160 through 1% water solvent casting. The treated particles were filtered and heat treated at 93 °C for 1 hr prior to mixing with the resin. The silane monomer added reactive functionality that chemically bound the particles to the matrix during the cure process.

2.1.3 Adherends

The adherends were constructed of 40 plies of Hexcel (Dublin, CA) unidirectional glass fiber/8551 epoxy prepreg. After fabrication, they were cut into lap shears $1.0 \times 4.0 \times 0.2$ in, with the fiber direction aligned along the long axis of the adherends.

2.1.4 Induction Unit

An induction unit operates by sending an alternating current through a conductive coil, which then generates an alternating magnetic field. This study used an Ameritherm NovaStar 1M (Scottsville, NY) induction unit, which operated at frequencies between 10 and 15 MHz. The magnitude of the magnetic field was adjusted by the "load power" (LP) setting. Increasing LP increased the amount of current that entered the coil and thus increased the magnetic field amplitude. The LP setting ranged from 0 to 1500 W, although this value did not correspond to the amount of power being dissipated by the induction coil or susceptor material. The magnetic field strength was also not necessarily linearly proportional to the LP.

2.1.5 Induction Coil

The coil used on the induction unit had a unique geometry, a solenoid designed specifically to process five lap shear specimens simultaneously (Figure 1). The coil was a three-turn solenoid fabricated from 0.3175-cm-diameter copper tubing. The solenoid was ~16.51 cm wide, 5.08 cm high, and 3.81 cm long. This geometry allowed simultaneous processing of five lap shear specimens simultaneously, decreasing processing inconsistencies. The bond areas of the lap shear specimens were placed next to one another, as shown in Figure 2.
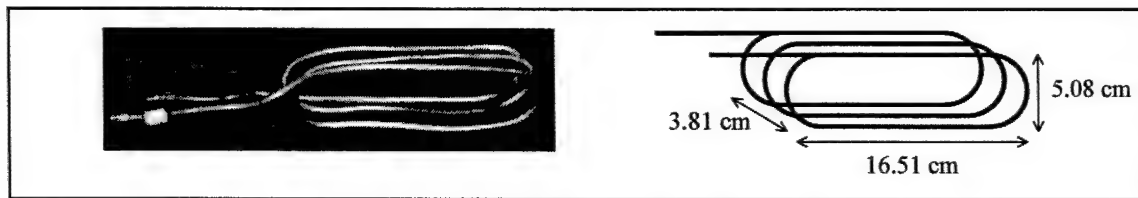
Figure 1. Photograph and dimensions of the three-turn solenoid induction coil.
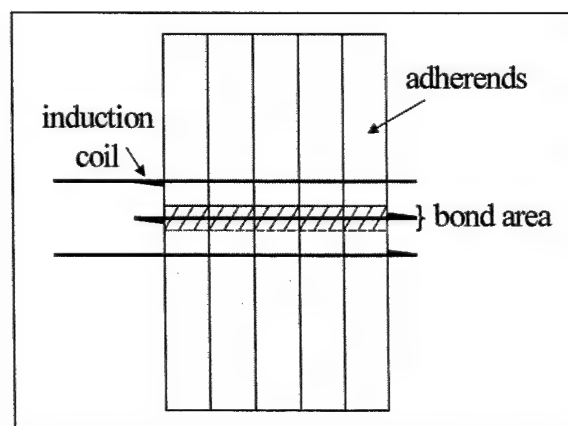
Figure 2. Location and position of the lap shear specimens during processing.

The operating frequency f of the induction unit was dependent upon the inductance of the coil and the capacitance within the circuit and is given approximately by

$$f = \frac{1}{2\pi\sqrt{(LC)}},$$

(1)

where L is the inductance of the coil and C is the capacitance within the circuit. The combination of system capacitance and inductance must be chosen to ensure that the operating frequency lies within the unit's rated range of 10–15 MHz.

Using an LCR meter, we measured the coil's inductance to be 1.04 μH. The Ameritherm unit was then configured to an internal capacitance of 100 pF, which, according to equation 1, should produce a frequency of ~15.6 MHz. Using this configuration, a frequency of 14.4 MHz was reported by the Ameritherm unit during processing.

2.1.6 Fiber-Optic Temperature Sensor

A thermocouple could not be used as a temperature sensor since it consisted of metallic wires which heated inductively. Additionally, because thermocouples use a voltage difference to measure temperature, the voltages induced by the alternating magnetic field can corrupt measurements. In order to obtain accurate temperature data, we used a fiber-optic temperature sensor manufactured by FISO Corp. (Quebec, Canada). The measurement zone of the fiber-optic temperature sensor was located at the end of the sensor and was ~10 mm in length (Figure 3).

Figure 3. Measurement zone of the
fiber-optic temperature sensor.

Figure 4 displays the geometry of the fiber-optic sensor in the lap shear bondline. The fiber-optic sensor's entire measurement zone was embedded at the center of the bondline. A groove was made in one of the lap shear surfaces so that the fiber-optic sensor was in direct
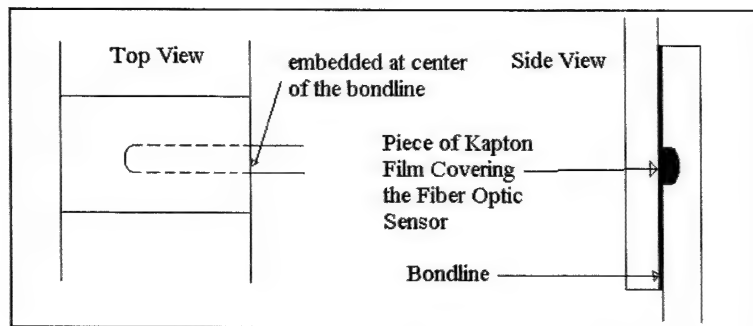
Figure 4. Top and side views of the position of the fiber-optic
temperature sensor within the adherend.

contact with the bonding surface. A layer of Kapton* film was placed between the fiber optic
sensor and the adhesive so that the sensor could be removed from the bondline once the
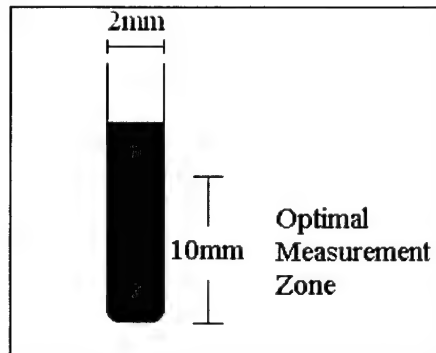experiment was complete. A single heating experiment was performed with the fiber-optic
temperature sensor, at an LP of 1500 W.

2.1.7 Cure Characteristics

Based on a series of isothermal experiments performed between 160° and 220 °C, a differential
scanning calorimeter (DSC) was used to analyze the cure characteristics of the epoxy. For all
experiments, the sample chamber with the reference was preheated to temperature prior to
staging the sample. The crossover point ($\Delta H = 0$) was used as the initial cure time (t = 0) in the
conversion analysis. A single fixed heating rate experiment (10 °C/min to 375 °C) was also
performed to assess the thermal degradation characteristics of the adhesive system.

2.2   Sample Preparation

2.2.1 Surface Preparation

Preparing the surface of the lap shears consisted of surface abrasion followed by cleaning.
Surface abrasion was accomplished by grit blasting with 50-μm aluminum oxide grit at 80 psi.
The bonding area was the only surface treated by abrasion. Once this was completed, the lap
shears were cleaned with deionized water and allowed to dry. The dry adherends were then
rinsed with acetone to remove any residual contaminants off the surface.

2.2.2 Adhesive Mixing

The magnetic particles were blended with the epoxy adhesive at a loading of 50% by weight
(~20% by volume). The mixing was done by hand until particle wet-out was achieved, followed
by dispersion in a high-speed mixer (7000 revs/s) for ~20 min. Care was taken to keep

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* Kapton is a registered trademark of DuPont.

temperatures below 70 °C during blending. After high-speed mixing, the mixture was placed in a vacuum chamber at full vacuum (25–28 mmHg) and 50 °C to degas for 16 hr.

2.2.3 Geometry of Bond

The adhesive was applied to the pretreated overlap area of both adhesives as an even coat ~0.0254 cm thick. Finger pressure was used to ensure intimate contact between the lap shears. Kapton tape was then wrapped around the bond area to ensure that contact was maintained during cure. A small amount of adhesive flash remained on both sides of the lap shears. Figure 5 shows the geometry for the lap shear.
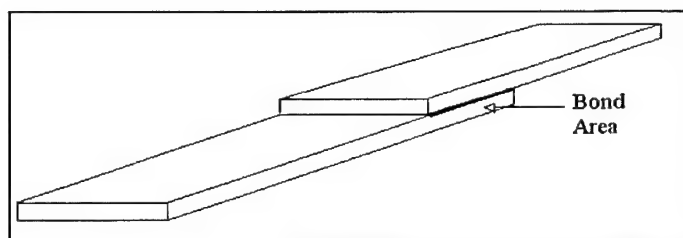
Figure 5. Lap shear geometry.

2.3 Processing

2.3.1 Oven-Cured Samples

To characterize baseline adhesive performance, two sets of five lap shears were oven cured at 175 °C for 1 hr. The first set contained neat adhesive, without magnetic particles. The second set contained the particle-loaded adhesive described in section 2.2.2.

2.3.2 Induction-Cured Samples

The lap shears were placed within the coil, on top of four sheets of Kapton film, to prevent electrical arcing from the coil. All five of the lap shears were processed simultaneously for each set of experimental conditions. They were located in the center of the coil (but resting on the lower coil windings), directly adjacent to one another, as previously described in section 2.1.5. Table 1 displays the powers and times for each set of samples cured in the induction field. Due to electrical arcing, the shortest 1500-W experiment was limited to 13 min, rather than 15 min.

Table 1. Power and times processed for the induction-cured samples.

Power Level (W)	Processing Times (min)		
500	15	30	60
1000	15	30	60
1500	13	30	

2.3.3 Mechanical Testing Setup

Lap shear strength measurements were performed using an Instron (Canton, MA) 4505 mechanical tester, with a 10-kN load cell. Figure 6 illustrates the testing geometry. The grip spacing for all experiments was 11.43 cm. Shimming tabs were placed against the adherends in the grips to minimize bending loads during testing.
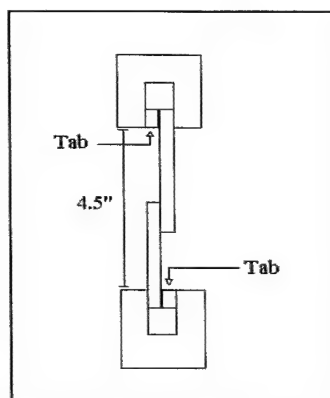
Figure 6. Geometry for the lap
shear test.

3. Results

3.1 Adhesive Kinetic Characterization

3.1.1 Particle Effect on Cure Kinetics

To determine whether the magnetic particles influenced the cure chemistry of the adhesive system, 180 °C isothermal DSC runs were performed on the uncured adhesive, both with and without magnetic particles. Figure 7 shows conversion as a function of time for the two systems, where full conversion is achieved when $\alpha = 1$. The conversion histories for the two systems were nearly identical. Therefore, as expected, the addition of the magnetic particles did not appear to impact the cure chemistry of the adhesive.

3.1.2 Cure Kinetics

To quantify the complete cure kinetics for the D1 system, isothermal runs on the DSC were performed using unfilled adhesive. The unfilled system cure kinetics should also represent the filled system, since the results of section 3.1.1 showed that the magnetic particles did not affect adhesive cure chemistry. Figure 8 shows the measured conversion histories for each temperature.

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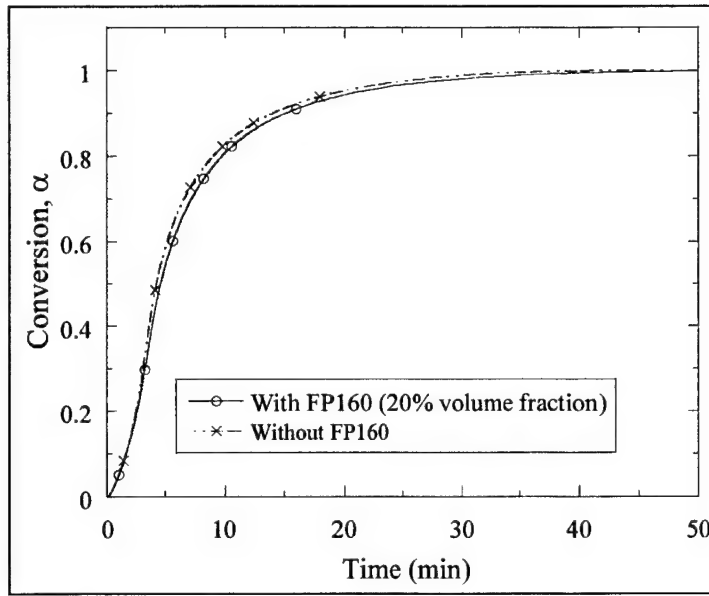
Figure 7. Conversion vs. time from 180 °C isothermal DSC
experiment on the D1 adhesive system with and without
magnetic particles.
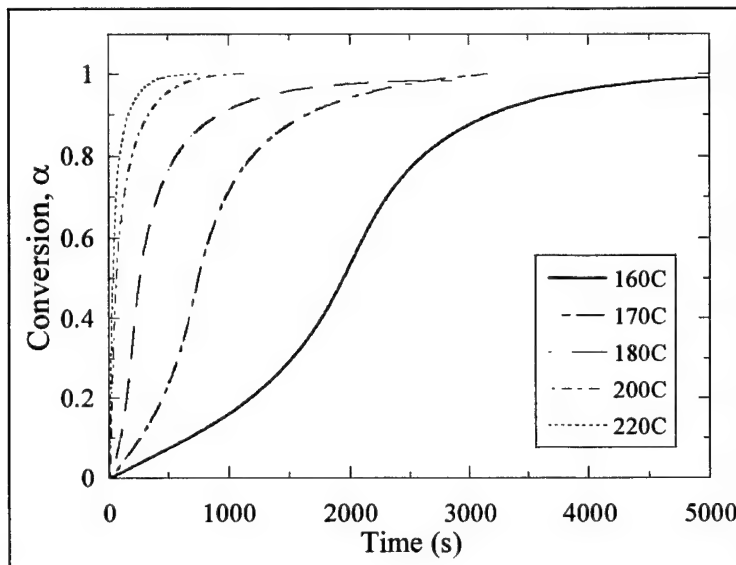
Figure 8. Conversion vs. time from isothermal DSC experiments for
the D1 adhesive system without magnetic particles.

Figure 8 shows that, as expected, cure time decreased as temperature increased. Table 2 lists the
time to reach 95% conversion for each of the isothermal runs, a reasonable benchmark for
effective cure. At 160 °C, full cure was approached in ~57 min, while only ~4 min were
required at 220 °C.

Table 2. Time for D1 adhesive system to reach 95% cure at various temperatures.

Temperature (°C)	Time to Reach 95% Cure (min)
160	57.1
170	36.4
180	23.1
90[a]	14.6
200	9.30
210[a]	5.90
220	3.74

[a]The 190° and 210 °C isothermal cure times have been interpolated using equation 2.

Figure 9 shows time to achieve 95% cure as a function of temperature. This data is well modeled by an Arrhenius equation as follows:

$$\alpha(T) = 0.236 \cdot e^{-1.29 \times 10^4 / RT} , \tag{2}$$

where α is conversion, T is temperature in kelvin, and R is the universal gas constant in joules/mole kelvin. This equation was used to interpolate the cure times for $T = 190°$ and $210 °C$ in Table 2.
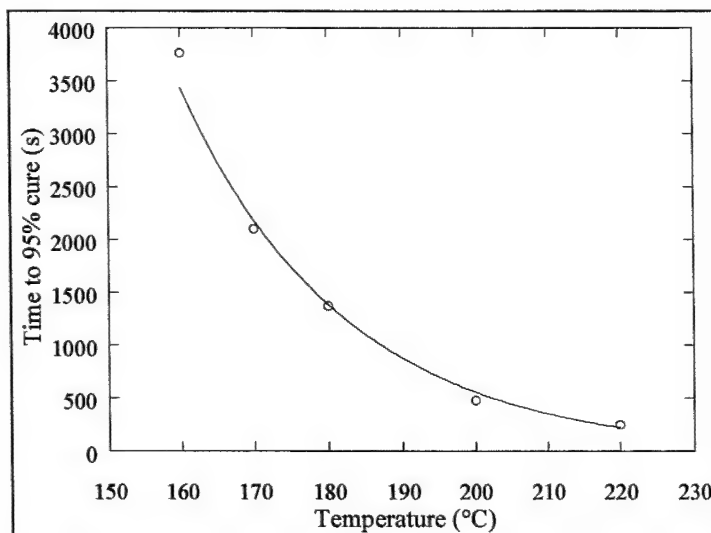
Figure 9. Time for the D1 adhesive system to reach 95% cure as a function of temperature.

3.1.3 Thermal Degradation Limits

In order to determine the thermal degradation limits of the adhesive system, a DSC run was performed on the unfilled adhesive (Figure 10). A heating rate of 10 °C/min was used up to 375 °C. For cure characterization in sections 3.1.1 and 3.1.2, the degree of cure was calculated
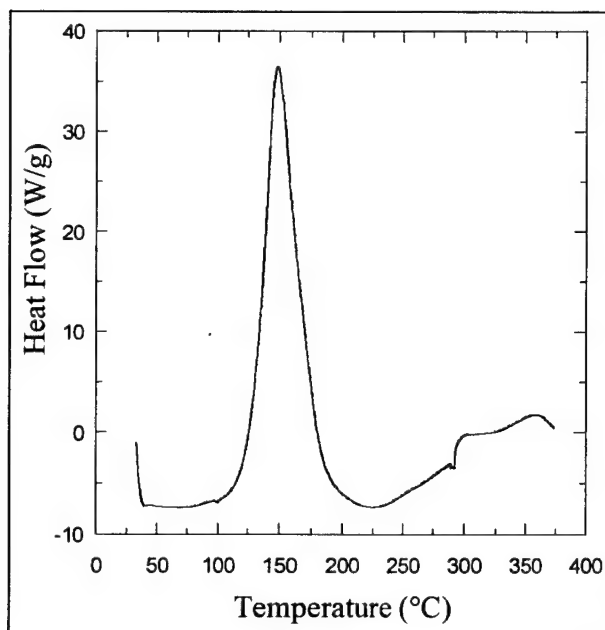
Figure 10. Heat flow as a function of temperature from
constant heating rate DSC experiment on
the D1 adhesive system without magnetic
particles.

based on the peak area between 100° and 220 °C. The additional heating generated above
250 °C could be due to continued or secondary curing of the adhesive system. Above 280 °C,
the sharp increase in heat flow indicated the occurrence of new reactions, which were likely
associated with thermal degradation of the adhesive. Figure 11 depicts a thermal gravimetric
analyzer (TGA) analysis of the unfilled adhesive system. The negligible weight loss beneath
250 °C indicated thermal stability beneath that temperature. This conclusion was consistent with
reports on the thermal stability of DICY-cured epoxies (15).

3.1.4 Thermal History During Induction Heating

A measurement of the temperatures reached within the adhesive was performed using a fiber-
optic sensor embedded within the bondline of the two adherends. Figure 12 depicts the
temperature reached within the bondline at 1500 W, with 50% weight fraction of magnetic
particles. The bondline temperature reaches equilibrium in ~5 min, and asymptotically
approached a value of ~210 °C. Once it reached this temperature, the adhesive dwelled at this
temperature, regardless of time exposed to the induction field. This temperature was well below
the expected adhesive thermal degradation temperature of 250 °C (section 3.1.3).

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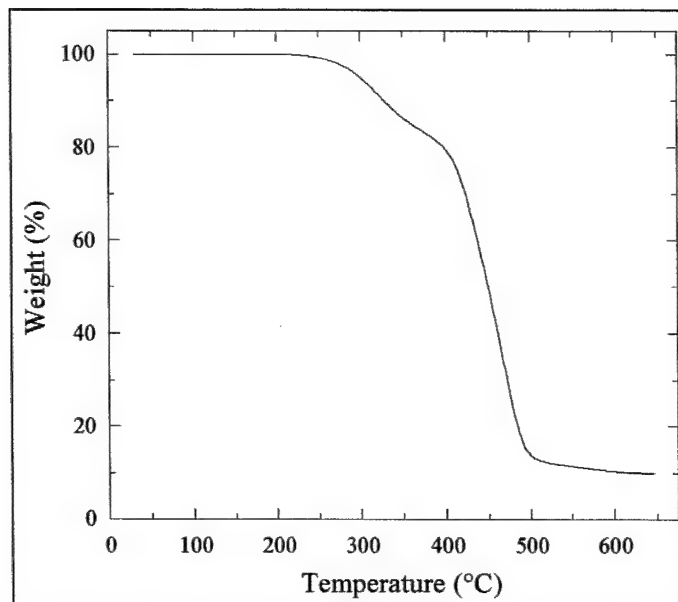
Figure 11. TGA measurement of weight loss as a function of temperature for the D1 adhesive system.
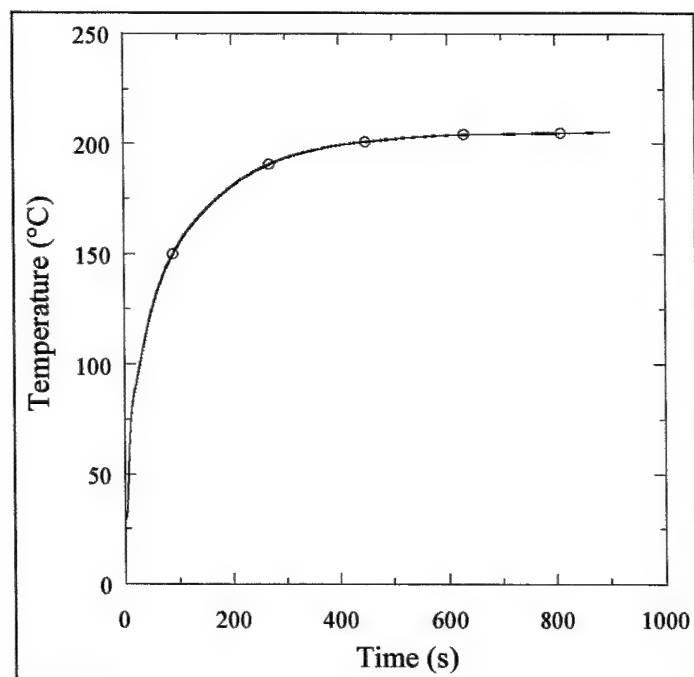
Figure 12. Heating history for the D1 adhesive system, with 50% weight magnetic particles, at a power setting of 1500 W.

We were unable to directly measure temperature histories at power levels of 500 and 1000 W because of equipment failure. However, previous experiments with a similar adhesive system with 50% weight FP160 magnetic particles achieved temperatures of 220°, 180°, and 165 °C at 1500, 1000, and 500 W, respectively (*11*). The heating rate changed only slightly for various power levels, with the time to reach the dwell temperature ranging from 5 to 7 min. We expect similar general heating behavior for the adhesive system used in this study.

3.2   Mechanical Properties

Table 3 shows the lap shear strengths for the oven-cured samples. The average mechanical strength of the oven-cured adhesive without the addition of the magnetic particles was 30.3 MPa. The average mechanical strength with the addition of the magnetic particles was 28.3 MPa, or a 6.5% decrease in mechanical strength. Therefore, the addition of the magnetic particles slightly diminished the mechanical strength of the adhesive system.

Table 3. Measured lap shear strengths for oven-cured samples.

Displacement at Failure (cm)	Length (cm)	Width (cm)	Load at Failure (N)	Lap Shear Strength (MPa)	Average (MPa)	Standard (MPa)
Thermal Cure Without FP 160, 175 °C for 1 hr (No Bondline Controls)						
0.371	1.23	2.63	603	38.9	30.2	5.41
0.296	1.42	2.65	378	24.4	—	—
0.327	1.37	2.64	424	27.3	—	—
0.356	1.44	2.55	473	30.5	—	—
0.306	1.36	2.62	468	30.2	—	—
Thermal Cure With FP 160, 175 °C for 1 hr						
0.317	1.37	2.60	419	27.0	28.2	1.38
0.368	1.37	2.66	440	28.4	—	—
0.382	1.38	2.57	468	30.2	—	—
0.259	1.38	2.62	445	28.7	—	—
0.312	1.34	2.66	415	26.8	—	—

Table 4 depicts the lap shear strengths for the induction-cured samples. All of the lap shear specimens displayed a cohesive failure. There was no correlation between location in the induction coil and the mechanical strength of the individual lap joints.

Table 4. Measured lap shear strengths for induction-cured samples.

Processing Power (W)	Processing Duration (min)	Displacement at Failure (cm)	Length (cm)	Width (cm)	Load at Failure (N)	Lap Shear Strength (MPa)	Average (MPa)	Standard (MPa)
500	15	0.066	1.39	2.59	123	7.93	4.88	2.78
—	—	0.034	1.33	2.64	48.0	3.09	—	—
—	—	0.061	1.37	2.64	101	6.49	—	—
—	—	0.027	1.37	2.60	31.2	2.01	—	—
500	30	0.332	1.24	2.63	317	20.4	18.8	2.29
—	—	0.434	1.41	2.61	320	20.7	—	—
—	—	0.302	1.38	2.64	315	20.3	—	—
—	—	0.234	1.48	2.71	265	17.1	—	—
—	—	0.258	1.41	2.57	244	15.7	—	—
500	60	0.329	1.35	2.64	383	24.7	23.7	1.02
—	—	0.300	1.37	2.61	368	23.7	—	—
—	—	0.330	1.25	2.60	365	23.5	—	—
—	—	0.288	1.36	2.62	379	24.5	—	—
—	—	0.318	1.38	2.59	343	22.1	—	—
1000	15	0.244	1.37	2.64	301	19.4	18.7	1.89
—	—	0.316	1.32	2.64	321	20.7	—	—
—	—	0.229	1.39	2.58	277	17.9	—	—
—	—	0.346	1.49	2.64	245	15.8	—	—
—	—	0.275	1.32	2.62	302	19.5	—	—
1000	30	0.240	1.34	2.67	259	16.7	19.5	1.72
—	—	0.350	1.30	2.63	305	19.7	—	—
—	—	0.269	1.36	2.59	303	19.5	—	—
—	—	0.362	1.36	2.67	326	21.0	—	—
—	—	0.439	1.27	2.63	322	20.8	—	—
1000	60	0.333	1.50	2.65	342	22.1	19.4	2.12
—	—	0.307	1.38	2.60	257	16.6	—	—
—	—	0.290	1.36	2.61	296	19.1	—	—
—	—	0.271	1.27	2.59	323	20.9	—	—
—	—	0.368	1.35	2.62	287	18.5	—	—
1500	13	0.331	1.30	2.73	269	17.3	17.7	1.60
—	—	0.244	1.26	2.62	309	20.0	—	—
—	—	0.245	1.35	2.62	251	16.2	—	—
—	—	0.220	1.34	2.64	269	17.4	—	—
1500	30	0.244	1.37	2.61	313	20.2	17.7	2.45
—	—	0.202	1.38	2.62	290	18.7	—	—
—	—	0.154	1.41	2.63	213	13.7	—	—
—	—	0.189	1.33	2.58	266	17.2	—	—
—	—	0.207	1.35	2.63	290	18.7	—	—

Figure 13 shows the average lap shear strength and standard deviation for each of the processing conditions. The maximum strength achieved by induction processing occurred at 500 W for 60 min. At 500 W for 15 min, the adhesive still possessed liquid-like properties (the flash was wet to the touch) and did not provide significant mechanical strength. At 1000 W, there was a slight improvement in mechanical strength between processing for 15 and 60 min. The same trend occurred for processing at 1500 W, where strength only increased marginally with increased processing time. This showed that at high power levels, further exposure to the induction field would not greatly enhance mechanical strength. All of the induction-cured specimens exhibited lower strength than the oven-cured specimens, with the 500 W for 60-min case reaching 84% of the strength of the filled, oven-cured samples.
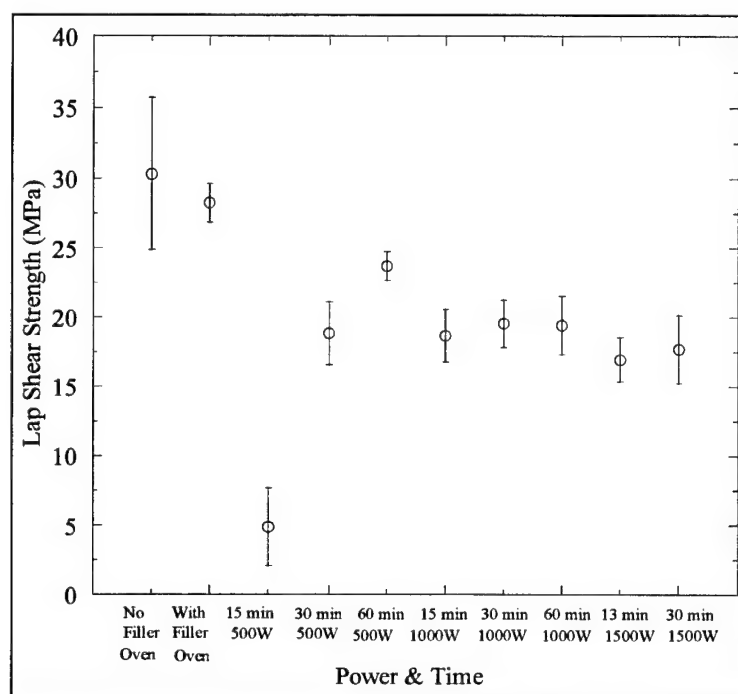
Figure 13. Average lap shear strengths for all processing conditions.

4. Analysis

4.1 Particle Effects

4.1.1 Particle Effect on Adhesive Cure Chemistry

The DSC measurements from section 3.1.1 demonstrate that the magnetic particles do not interact chemically with the D1 adhesive. This result is not surprising since they are stable oxide particles that do not give off any gases or interact with the organic chemistry of the adhesive.

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4.1.2 Particle Effect on Bond Mechanical Performance

Under comparable oven-cured conditions, the presence of magnetic particles resulted in a slight decrease in adhesive mechanical properties, as compared with the unfilled system. However, the relatively large scatter in the data for the unfilled system makes it difficult to draw conclusions with high confidence. Note that if the specimen with the highest lap shear strength in the unfilled sample is discounted, the average load at failure between the filled and unfilled D1 adhesive is equivalent within statistical error. To reduce scatter in future efforts, more emphasis needs to be placed on maintaining constant bondline thickness and adherend thickness.

4.2   Analysis of Strength Trends

Obviously, thermal histories play a crucial role in the curing of the adhesive. In general, induction processing causes the adhesive to heat rapidly and then dwell at some relatively stable temperature (refer to Figure 12). The heating ramp rate only alters slightly between various power settings but, in all cases, approaches its dwell temperature within 5–7 min. Therefore, the only effect of time at a constant power, after the initial 5-min ramping period, is further time spent at the dwell temperature. The major effect of the power is the determination of the final dwell temperature, with higher power levels producing higher dwell temperatures.

Based on the DSC run from section 3.1.3 and temperature histories from section 3.1.4, we can assume that thermal degradation is probably not occurring within the adhesive. According to the DSC run, the onset of degradation begins around 280 °C, while the force integration staff officer (FISO) data indicates a maximum temperature of 210 °C in the adhesive during induction processing. Also, the 1000- and 1500-W cases show no decrease in mechanical strength with longer exposure to the induction field. These two observations imply that thermal degradation of the adhesive is not occurring.

Figure 13 shows that at 500 W, increasing induction exposure time leads to increasing mechanical properties. After processing at 500 W for 15 min, the adhesive appears to be liquid-like, corresponding to an incomplete degree of cure. This observation likely indicates that the increase in strength noted with increasing exposure time at 500 W is due to increasing degree of cure. In contrast, the 1000- and 1500-W samples show only minor improvements in strength for process times greater than 15 min. Therefore, it is likely that the adhesive was mostly cured after 15 min at those higher power levels. Postprocess DSC analysis needs to be performed to verify all of these hypotheses.

The maximum strength achieved for each power level appears to decrease with increasing power, although the trend is not striking. These results could indicate that the difference in cure kinetics or toughening kinetics at higher temperatures directly leads to decreasing adhesive performance. Further analysis and experimentation are needed to confirm this hypothesis.

The best adhesive bond performance is achieved at a power level of 500 W and an exposure time of 60 min. Under these conditions, the lap shear strength of the induction-processed sample reaches 84% of the filled, oven-cured system lap shear strength.

5. Conclusions

5.1 Magnetic Particle Susceptors for Induction Heating

We have shown that induction heating can achieve thermal powers necessary for thermal curing of adhesives, enabling both rapid heating and high dwell temperatures. This achievement is not trivial and has been made possible only through recent advancements in induction processing equipment. Specifically, the commercial availability of high-frequency (greater than 10 MHz), self-tuning induction power supplies has allowed for efficient energy transfer to magnetic susceptor particles.

The main advantage of induction heating arises from utilization of the Curie temperature of the magnetic susceptor particles. With a well-defined Curie temperature, a magnetic powder-loaded adhesive system will be thermally self-regulating. Curie limiting is only partially utilized in our study. The magnetic particles in our study do not possess a well-defined Curie temperature, so that dwell temperature is highly dependent on induction field strength. Furthermore, the dwell temperature is not a true dwell, as temperatures slowly but continuously increase during dwell. Better heating performance and control could be achieved by using magnetic materials developed specifically for Curie temperature-controlled processing applications (7).

5.2 Rapid Curing of Adhesive Systems

The choice of an adhesive system is critical for effective utilization of induction heating. The epoxy adhesive system used in this study is based on a commercial formulation designed to cure thermally in ~1 hr. Thus, it is sensitive to time-temperature profiles, which affect network formation and rubber phase development. The adhesive system evaluated is not designed to cure inside of 15 or 30 min. Induction heating would serve as an optimum type of curing for an adhesive system without a dependency on the heating rate (e.g., free radical initiated). These types of adhesive systems would best utilize the advantages of induction heating over other types of thermal curing.

To fully demonstrate induction heating as method for rapid adhesive processing, a comprehensive analysis must be undertaken of a heating rate insensitive adhesive system. We

are preparing to do this with additional formulations of adhesive systems, which have the preformed rubber particles added a priori. Eliminating the phase separation issue from the performance will allow us to address issues associated with rapidly formed thermoset network structures independently.

6. References

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